REPORT R-1792

A VACUUM PATH X-RAY DIFFRACTOMETER FOR PRECISION RELATIVE LATTICE PARAMETER MEASUREMENTS ON SINGLE CRYSTALS

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ABSTRACT

A vacuum path X-ray diffractometer designed for lattice parameter measurements on single crystals is described. It is an instrument of high precision approaching 1 part in 10^6 or better, and is also capable of moderately high accuracy of approximately 1 part in 10^5. The instrument is conceptually a variation of an earlier design but various factors are introduced to facilitate convenient experimental conditions. Scanning is automatic and provision is made on the instrument for a large working volume to accommodate auxiliary environmental equipment for the specimen. The mounting and measurement procedure is described and a sample calculation of lattice parameter with associated accuracy and precision is given. A discussion of the errors encountered in high precision measurements of this type concludes the report.

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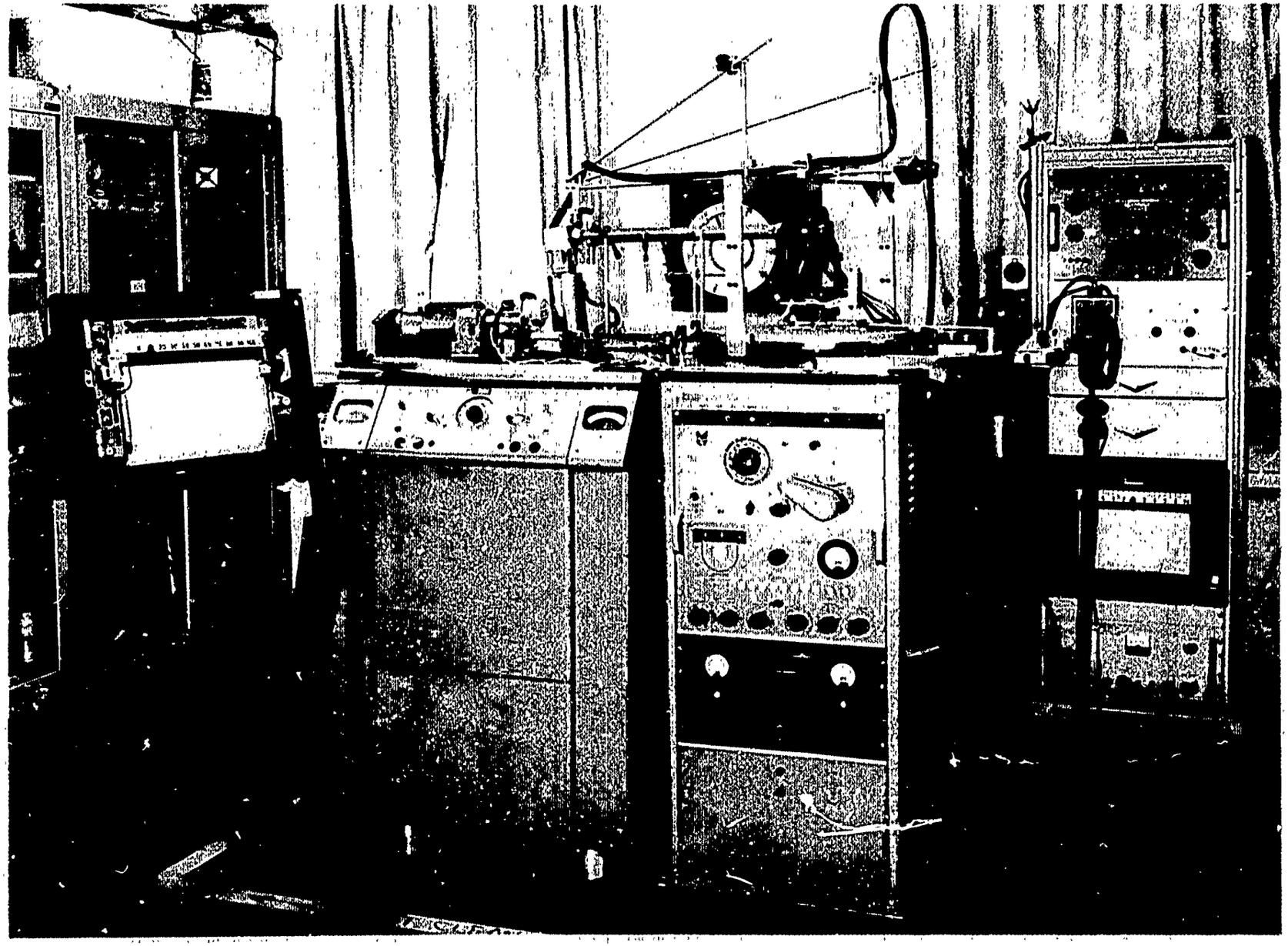

Frontispiece. X-ray Diffractometer

INTRODUCTION

Construction of the X-ray diffractometer shown in the frontispiece was undertaken with some specific measurements in mind, primarily comparison lattice parameter (a_o) measurements on uncolored alkali halides and similar crystals with various concentrations of color centers. High temperature measurements of a_o on various metals and alloys were also contemplated. Deviations from dilatometric measurements were of interest. For these reasons prime consideration was given to maximizing precision and sensitivity with secondary emphasis on absolute accuracy. Both the accuracy and precision obtainable may be considered a function of the angle of diffraction and, therefore, the instrument has been designed for measurements at high angles of the back reflection region in order to enhance these factors. Throughout the construction an attempt was made to utilize existing laboratory components or surplus parts to minimize costs.

For versatility, provision was made for a large working volume. The diffractometer has been used with specimens with linear dimensions from a millimeter to about an inch, but much larger samples can be mounted. The large working volume also enables various high and low temperature sample chambers to be mounted easily. The sample can also be mounted in a vacuum or controlled atmosphere to eliminate oxygen contamination at high temperatures or to inhibit deterioration of the surfaces of the hydroscopic crystals.

The diffractometer is conceptually a variation of Bond's[1*] diffractometer, but differs from it considerably in construction and working arrangements. Scanning is automatic with this machine and considerable operator time and trouble are saved thereby.

DESCRIPTION OF APPARATUS

Goniometer

The beam originates at the point focus of a CA-7H high intensity X-ray tube see (a) (figs. 1 and 2) and impinges on the mounted specimen surface

*See References.

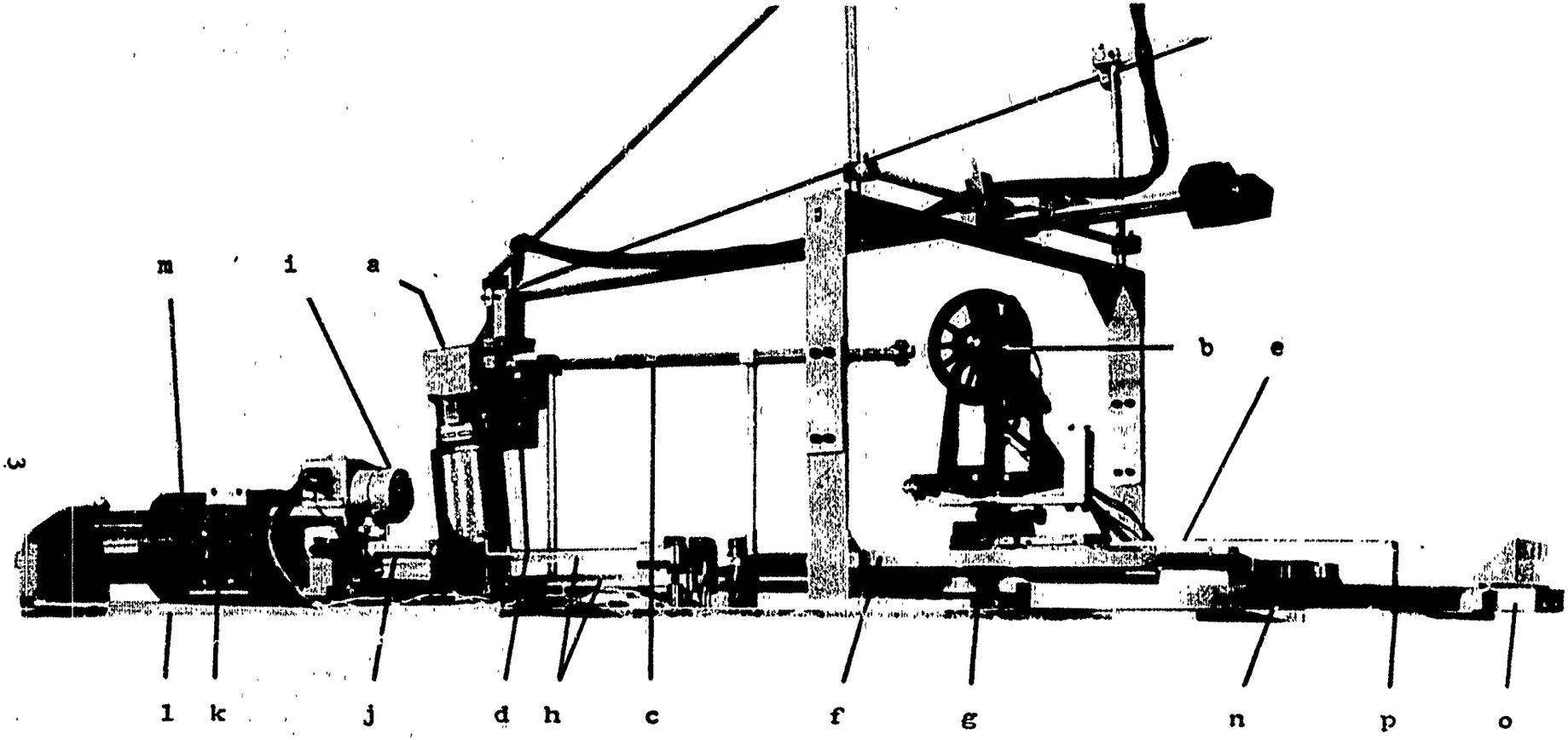

Figure 2. Diffractometer Side View

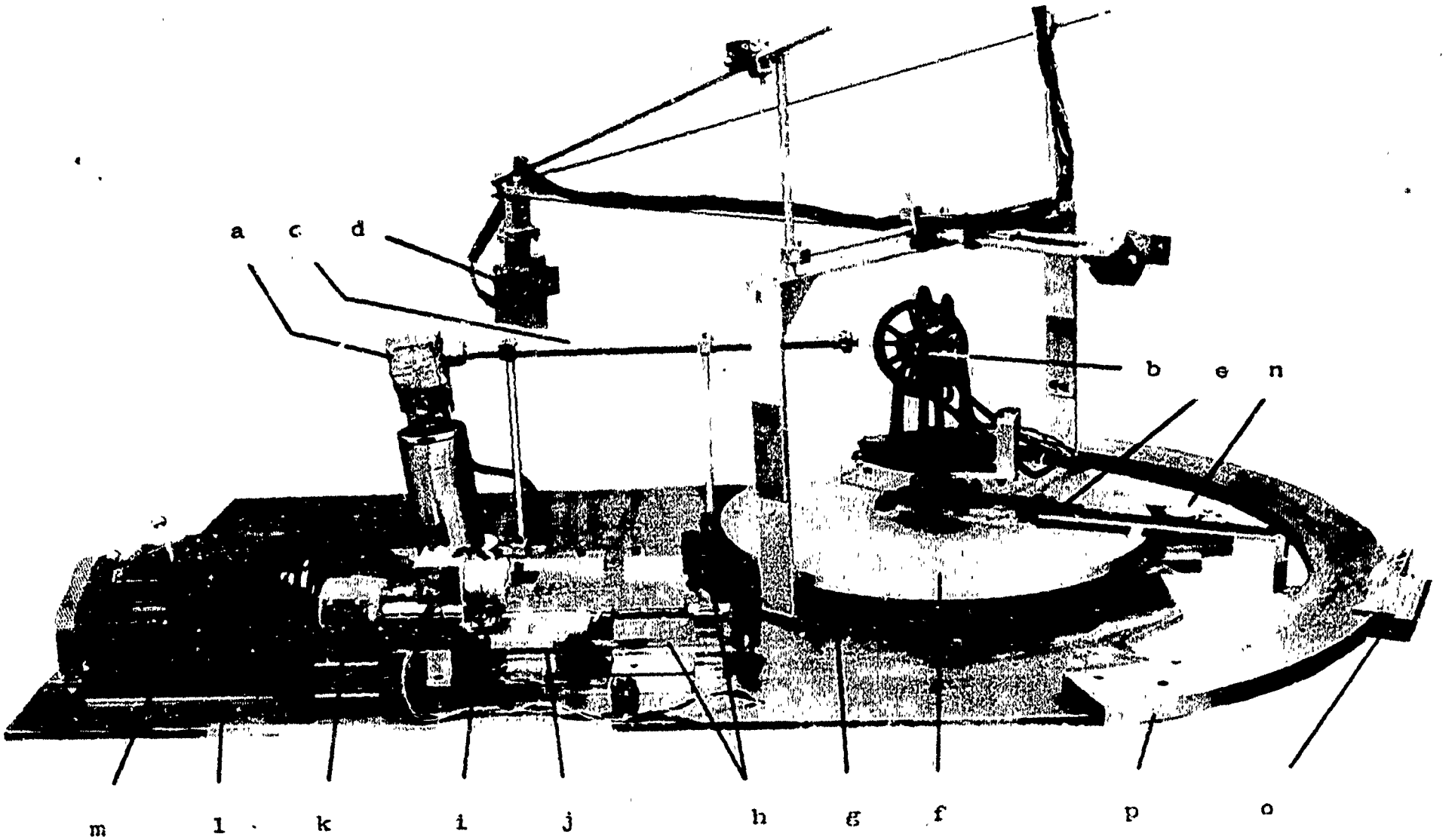

Figure 1. Diffractometer Top View

(b) 75 cm away. The tube itself is tilted through the take-off angle of 3° (from the vertical) so all other apparatus can be horizontal. The collimator (c) is an evacuated tube with the X-ray tube anode itself serving as the initial pinhole.[2] The exit pinhole, 65 cm away, has a 25 millimeter diameter, resulting in a circular beam with divergence of 7'. The diffracted beam also travels through a large evacuated tube (shown in place in frontispiece only) with mylar windows, and enters the window of the proportional counter (d) which travels at the end of a 80 cm boom. Since the angle of rotation is measured by an arm (e) attached to the crystal, there is no need for any slit system on the window of the counter, and any vibration which might appear (very little is actually present) at the counter support will not affect the accuracy of the measurements.

The goniometer consists of two discs (f, g) coaxially mounted in the horizontal plane and rotated by steel bands (h) attached to their perimeters. The discs are accurately machined with a diameter ratio of 2:1. The specimen chamber (b) sits on the center of the upper (larger) disc.

Rotation is initiated by a small timer motor (i) which turns a precision grating-ruling screw (j) at various speeds through a 10 speed gear box (k). As the screw turns, the nut on the ruling screw pulls two steel tapes (h) at equal and constant speed. As the rotation rate of the two discs varies inversely as their diameters, the smaller one (to which the counter is attached by a boom) turns at twice the rate of the larger. The precision rotation can thus be varied from 0.48 seconds of arc per second to 4.8 seconds of arc per second in 10 steps. Coarse rotation is supplied by moving the timer motor and ruling screw system along a track (l) with an adjustable speed motor (m), allowing equivalent rotational speeds in either direction from zero to about 10° per second. "Negator" band springs attached to the discs supply back torque.

Angles are read directly on a large vernier (o) and scale (p). The scale is mounted coaxially with the two discs and the vernier is attached rigidly to the specimen chamber.

Specimen Holders

The goniometer will accommodate specimen holders with diameters up to 10" and unlimited heights. The beam height is variable within wide limits but the normal height is 26 cm from the working surface.

For high temperature work quartz chambers with mylar windows have been used. The specimen is fastened to a quartz rod in the chamber with asbestos putty. The chamber and concentric furnace are mounted on a three circle goniometer with provision for forward and backward movement, constructed from a surplus surveyor's transit. The specimen face is placed on the center of rotation, first by optical means and then by comparison of X-ray photographs taken at the Bragg position at either side of the zero position. These furnaces have been used successfully to 1000° C.

For low temperature work quartz designs are being tested which will allow us to grow crystals of various rare gases in situ, align these and proceed with measurements.

For work near room temperature water-heated furnace designs have been used which are capable of controlling the specimen temperature to ± 0.02° C. The furnaces accommodate specimens of lateral dimensions up to 1/2" square and any thickness. Thin specimens may be irradiated from the rear (for coloring purposes) by an auxiliary X-ray tube while still mounted for measurement or even during the measurements themselves. These furnaces are also based on two and three circle goniometers derived from surplus surveyors transits, and are devised so that the specimen is always mounted on the center of rotation.

MEASUREMENT PROCEDURE

Using a "slide rule" nomogram designed to solve the Bragg equation, the plane with reflection nearest to 88° theta (the maximum allowable on the instrument) is chosen and the appropriate X-ray tube mounted. The crystal face is made parallel to the plane of interest by cleaving, spark cutting, or some other appropriate means, after orientation by use of Laue photographs if necessary. The crystal is then mounted on the center of rotation of the crystal holder and goniometer by optical means.

The proportional counter is moved in on its boom until it is quite close to the crystal. A large angle is now subtended by the counter with respect to the crystal and the diffracted beam will enter some portion of the counter window when the crystal is set at the Bragg angle. The crystal is rotated independently of the counter until it is at the approximate

Bragg angle using the visible crystal face and the counter output as guides. The counter is then gradually moved back to its farthest position making adjustmens on the counter and crystal as necessary to keep the diffracted beam within the window. This process rarely takes longer than 10 or 15 minutes. With the counter locked in position, a half-millimeter horizontal slit is put into its window and the crystal "tilt" adjusted so that the diffracted beam is maximized. This assures that the incident beam and the two diffracted beams (on each side of the "zero" position) are in the same (horizontal) plane. A one-quarter millimeter error in height at the counter corresponds to a one minute misalignment of the crystal and a one-half second systematic error in angle theta. The diffracted beam diameter at this distance is around half a millimeter, depending on crystal perfection. A series of pairs of pictures is now taken with dental film mounted at the counter window, one each at the Bragg peak positions on each side of the X-ray tube. The crystal holder is moved backward or forward, as necessary, until the pictures of a given pair are identical with respect to spot substructure, indicating that the crystal is exactly on the center of rotation. Exposure time can be as little as 10 minutes (KCl; (800) plane; Cu $K\alpha_1$ radiation; 20 ma, 45 KV).

To determine the angle between the two diffraction peaks, a slow ($\sim$1 second of arc per second) scan is made through the α_1 peaks on each side of the X-ray tube, always scanning in the same direction. The vernier is read five or ten times during the scan near each peak. The scan is so slow that movement can barely be detected. A pen in the margin of the recorder records when the readings are made opposite the intensity trace. The so-called "mid-chord peak" is termined geometrically by extrapolating the line connecting the mid-points of the horizontal chords drawn at various fractions of the peak intensity until it intersects the line profile. Since this line is virtually vertical above 40% of peak intensity, and the upper part is used, the "mid-chord peak" coincides with the true peak. By measuring the distances between this peak position and the margin indications of vernier readings, a peak determination can be made (assuming constant angular movement) for each vernier reading. Small deviations, due to slip stick in the rotating discs or intensity variations, are smoothed out statistically. If desired, step scanning can also be made for even greater accuracy.

With this diffractometer, the angle φ between the two symmetrical settings of the crystal at positions of diffraction is measured instead of the Bragg angle θ. From geometrical considerations: $2\theta = 180^\circ - \varphi$.

The lattice parameter a_o can be calculated from the Bragg equation:

$$\lambda = 2d \sin\theta = 2 \frac{a_o}{f(h, k, l)} \sin\theta$$

or

$$\lambda = 2d \cos \varphi/2$$

where

λ = the wavelength of the incident X-rays

θ = the angle of incidence

d = the distance between the diffracting planes

a_o = the lattice parameter

$\varphi = 180° - 2\theta$

and

f(h, k, l) is a suitable function of the Miller indices (which $= (h^2 + k^2 + l^2)^{1/2}$ for a cubic material like Al or KCl).

POTASSIUM CHLORIDE MEASUREMENTS

Measurements on potassium chloride (KCl) single crystals were begun in an attempt to correlate changes in lattice parameter with point defect concentration produced by additive coloration. These changes were masked initially by variations in a_o due to mounting strains and strains due to the weight of the crystal itself. Differences as large as 0.00025 Å (4 parts in 10^5) were found on a single specimen by translating it and determining the lattice parameter at different points on the surface. From photographic evidence it is estimated that the mosaic block size was such that the X-ray beam irradiated about 10^5 blocks and, therefore, the differences observed would not be due to mosaic block variations.

In spite of this difficulty, it will be instructive to make a sample calculation from a typical run on a crystal.

The single crystals were cleaved parallel to a cube face (the (100) plane) and produced 8th order peaks which were slightly skew but symmetrical about the incident beam. The spectral half width (i.e., that due to the finite half width of the incident beam) is 5.7' at this angle, and the peaks were found to have a half width, L + R, (see fig. 3) of six to eight minutes. A typical peak had a peak to background ratio of 50 to 60 with a peak counting rate of about 800 cps.

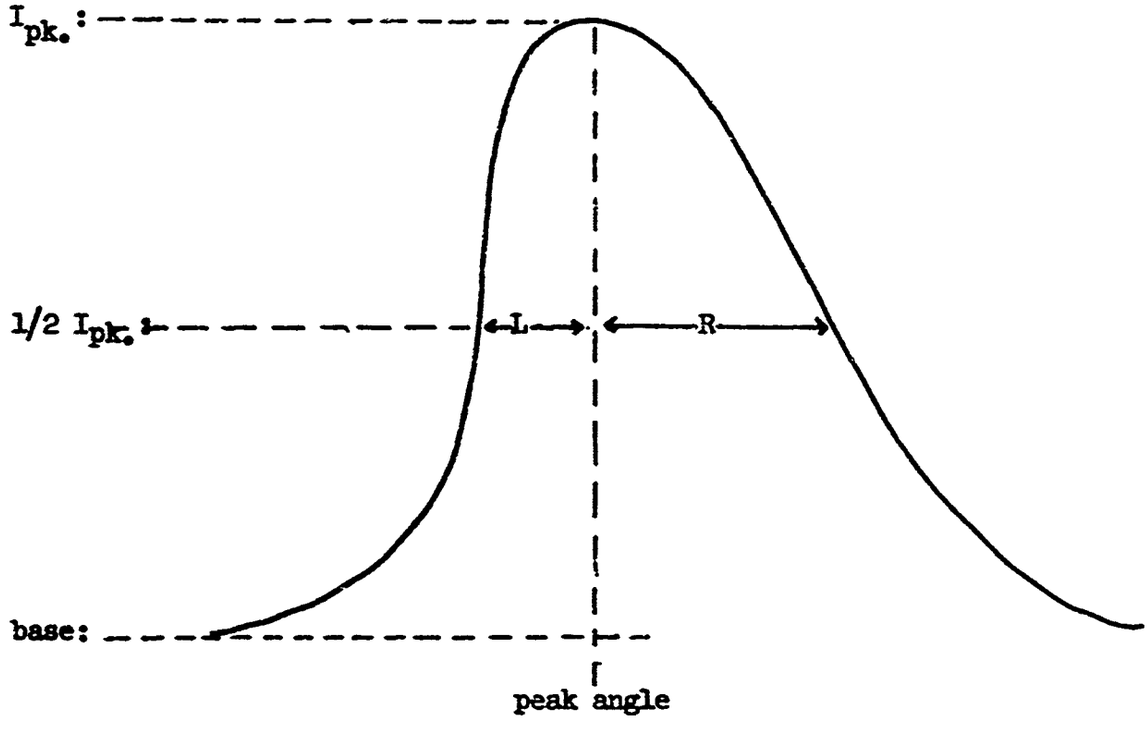

Figure 3. Representative Line Profile

The asymmetry index R/L (see fig. 3) varied from 1.00 to 1.07. The average angle φ between the peaks, calculated after ten runs, was found to be 23° 25' 40.5" ± 1.8". The corresponding value for the average Bragg angle is $\overline{\theta}$ = 78° 17' 9.8" ± 0.9". This internal precision is the standard deviation of the average value $\sigma_{\overline{\theta}}$. The standard deviation for any measurement σ_{θ_i} = ±2.8", and the Student's 90% confidence level is ±1.6" (θ). Corrections are as follows:

$$\theta = 78^\circ\ 17'\ 9.8'' \pm 0.9''$$

Lorenz-polarization correction: +0.5"

$$\theta \text{ corrected} = 78^\circ\ 17'\ 10.3''$$

Using

$$\lambda = 1.537400 \pm 1 \text{ p.p.m. kX.U.} \times 1.002057 \pm 5 \text{ p.p.m. Å/kX.U.}$$
$$= 1.540563 \pm 5 \text{ p.p.m. Å (peak)}^3,$$

we find:

$$a_o' = 6.293215 \text{ Å}$$

refraction correction: 1.0000077
axial divergence correction: 1.0000004

$$a_o = 6.293266 \text{ Å} \pm 0.0000057 \ (25.0^\circ \text{ C})$$

(0.9 parts in 10^6)

Wychoff[4], citing several investigators, gives for the potassium chloride lattice parameter:

a_o (Wychoff): 6.29294 Å (25° C) (as given)

a_o (Wychoff): 6.29321 Å (corrected to more recent value of wavelength)

a_o (present value): 6.293266 Å (25.0° C)

est. error: .00006 Å or 1 part in 10^5

The two calculations given above are illustrative of the accuracy and precision of which the instrument is capable.

ERRORS

Differentiating the Bragg relationship leads to the equation:

$$\frac{\Delta a_o}{a_o} = \frac{\Delta\lambda}{\lambda} - \cot\theta\,\Delta\theta$$

or

$$\frac{\Delta a_o}{a_o} = \frac{\Delta\lambda}{\lambda} - \tan\frac{\varphi}{2}\frac{\Delta\varphi}{2}$$

Neglecting $\Delta\lambda/\lambda$ for the moment it can be seen that $\Delta a_o/a_o$ approaches zero as θ approaches 90°. Therefore, a high precision measurement can be made even if the angle cannot be measured accurately as long as the measurement can be made on a diffraction peak very close to $\theta = 90°$. This is the raison d'etre of the back reflection method.

The work on this section is incomplete but has been taken to a point where the information presented, plus standard references[5, 6, and 7], can be used to provide the worker with sufficient information to use the machine to its fullest capabilities. Although there are several semi-exhaustive analysis of errors encountered in the literature on powder work, there is no compilation of the type given here for single crystal work on this type of instrument.

A list of errors considered follows in outline form with comments. The instrument is designed to eliminate some of the major common causes of errors. These are listed in square brackets. Those errors marked with an asterisk are systematic and, therefore, affect the absolute accuracy only and do not apply to relative measurements. Those indicated to be negligible were determined so by calculation or measurement in the laboratory.

	ERRORS	ERROR COMMENTS
Due to Physical Effects:		
A-1	*wavelength uncertainty	5 p.p.m. if peak values used for both λ and θ (3, 5)
A-2	*/dispersion/	not analyzed; in combination with errors A-9 and B-1 causes asymmetry of line shape and therefore affects center of gravity rather than the peak position which we use
A-3	*anomalous dispersion	negligible with our specimen materials (absorbtion edge in specimen must be near incident radiation wavelength for effect to be appreciable)
A-4	*/white radiation/	causes asymmetry; eliminated by pulse height analysis
A-5	*refraction	correction: +7.7 parts in 10^8 for KCl (800); (1)
A-6	*Lorenz	correction of +0.5" for KCl (800); (1, 5, 6)
A-7	*polarization	included in A-6
A-8	intensity fluctuation	negligible
A-9	*/absorbtion or specimen transparency/	see A-2
A-10	*non-infinite crystal thickness	causes asymmetry if significant, but completely negligible

	ERRORS	ERROR COMMENTS
Arising from Geometry:		
B-1	*/flat specimen (horizontal divergence)/	see A-2
B-2	*axial (vertical) divergence	correction 4 parts in 10^7 for our collimator (1); causes asymmetry
B-3	*source and slit width	causes only peak spreading; no error
B-4	*Eberhardt effect (peak overlapping)	$\ll$1"; neglected
B-5	*/filters, monochromaters/	not used
Inherent in Instrument:		
C-1	*scale accuracy	affects both accuracy and precision although latter very small; not tested in detail but estimated accuracy is 1 part in 10^4 or better
C-2	*vernier accuracy	up to 15" $\Delta\theta$, but negligible if care taken to avoid ends
C-3	*/2:1/	better than 1 part in 10^3 but not pertinent
C-4	/backlash/	eliminated by one way scan
C-5	*scale not centered on axis or rotation	negligible
C-6	*waveform of X-ray voltage	not analyzed
C-7	*/counter window/	
C-8	non-linear counter response	negligible if care taken to avoid center of counter

	ERRORS	ERROR COMMENTS
Inherent in Instrument: (Cont'd)		
C-9	coincidence counts	no error; only flattens peak
C-10	*/mylar windows/	any refraction correction eliminated by symmetry (non-uniformity of response because of segregated high-atomic-number impurities is negligible)
C-11	mechanical fluctuations	negligible
C-12	collimator movement	negligible
C-13	focal spot shifting	negligible
C-14	thermal strains in scale	<1"; long term maximum may be larger
C-15	voltage fluctuations in counter and generator	negligible
Alignment Errors:		
D-1	*/zero setting/	
D-2	*/beam asymmetrical about axis of rotation/	
D-3	*beam not perpendicular to axis of rotation	$\pm 1'' \Delta\theta$
D-4	*/crystal surface not coincident with axis of rotation/	
D	*crystal surface tilted, i.e., zone axis not perpendicular to axis of	$< \pm 0.5'' \Delta\theta$

	ERRORS	ERROR COMMENTS
Measurement Errors:		
E-1	*/peak vs. center of gravity/	not applicable because peak values of both wavelength and diffraction maxima used
E-2	*/time constant of electronics/	eliminated by very slow scan
E-3	geometrical choice of peak	$\pm 1.5''\ \Delta\theta$, in one run, but minimized by statistics
E-4	reading error, i.e. $\Delta\varphi$	$\pm 1.5''\ \Delta\theta$, in one run, but minimized by statistics
E-5	slipstick	$\pm 4''\ \Delta\theta$, in one run, but minimized by statistics
E-6	rotation not constant	negligible compared to E-5 above and completely masked by it
Due to Specimen:		
F-1	specimen impurities	not considered
F-2	specimen temperature control	$\ll \pm 1''$
F-3	mosaic structure	not pertinent if blocks much smaller than beam area as is usually the case
F-4	X-ray damage	negligible at measuring levels
F-5	mounting and other strains	largest error; can be up to $\pm 20''\ \Delta\theta$
G-1	*/extrapolation to $\theta = 90°$/	not applicable with this method; errors eliminated by other methods

SUMMARY

An X-ray diffractometer capable of high precision in the measurement of relative lattice parameters has been described. With this instrument it is possible to measure lattice parameters with an accuracy of a few parts in 10^5. Variations of lattice parameters approaching 1 part in 10^6 or better can be detected with nearly perfect crystals. Measurements have been made with some alkali halide crystals and with aluminum single crystals. Calibrations to improve the absolute accuracy are now in progress because of anticipated work on solid rare gases.

ACKNOWLEDGMENTS

We should like to acknowledge A. Stott for his helpful suggestions and thank D. Stango for his help in the laboratory.

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